

METHOD AND DEVICE FOR PRODUCING SILICON CARBIDE SINGLE CRYSTAL

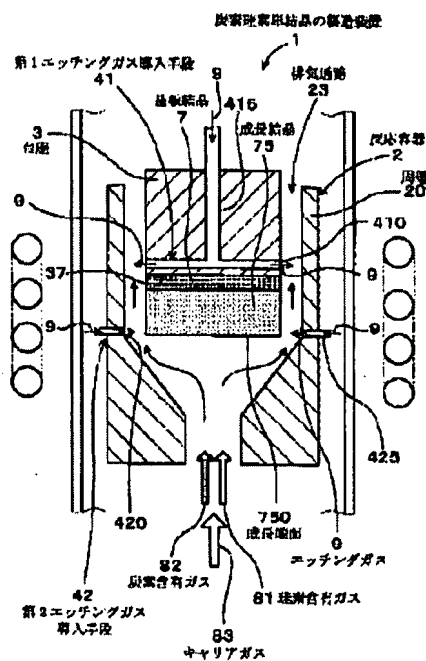
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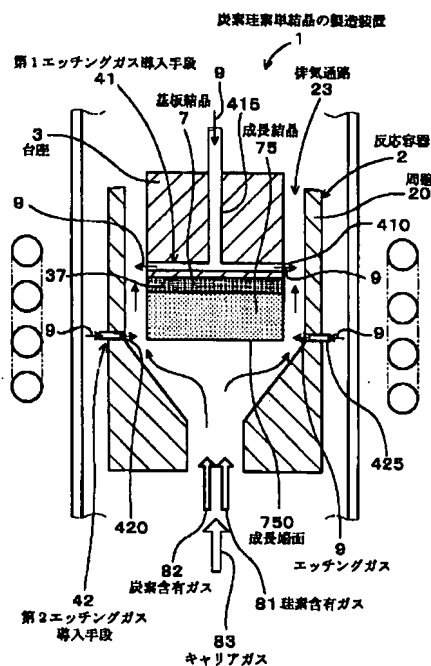
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PROBLEM TO BE SOLVED: To provide a method and a device for producing a silicon carbide single crystal, by which deposition of the silicon carbide single crystal at inappropriate places other than the growth end surface of the growing crystal can be suppressed, and the growth of the growing crystal for a long time is possible, and a manufacturing device for the silicon carbide single crystal. **SOLUTION:** A pedestal 3 for supporting a substrate crystal 7 formed from a silicon carbide single crystal is placed in a reaction vessel 2 having a cylindrical peripheral wall 20. The silicon carbide single crystal is grown on the substrate crystal 7 by supplying a silicon containing gas 81, a carbon containing gas 82 and a carrier gas 83 toward the substrate crystal 7 supported by the pedestal 3, and at the same time, the excess silicon containing gas 81, the excess carbon containing gas 82 and the excess carrier gas 83 are discharged through between the pedestal 3 and the peripheral wall 20. An etching gas of etching silicon carbide is introduced to the neighborhood of the periphery of the interface 37 of the substrate crystal 7 and the pedestal 3 and the neighborhood of the periphery of growing end surface 750 of growing crystal 75 of the silicon carbide single crystal grown on the substrate crystal 7.

(図1)





【特許請求の範囲】

【請求項1】 筒状の周壁を有する反応容器内に、炭化珪素単結晶よりなる基板結晶を支持する台座を配置し、該台座に支持された上記基板結晶に向けて珪素を含有する珪素含有ガスと炭素を含有する炭素含有ガスを供給して上記基板結晶上において炭化珪素単結晶を成長させると共に、上記台座と上記周壁の間を通過して余剰の上記珪素含有ガス及び上記炭素含有ガスを排出する炭化珪素単結晶の製造方法において、上記基板結晶と上記台座との境界面の外周近傍と、上記基板結晶上において成長した炭化珪素単結晶である成長結晶の成長端面の外周近傍に、炭化珪素をエッチングする効果のあるエッチングガスを導入することを特徴とする炭化珪素単結晶の製造方法。

【請求項2】 請求項1において、上記基板結晶と上記台座との境界面の外周近傍においては、上記台座の内部から外部へ向けて上記エッチングガスを吐出することにより導入することを特徴とする炭化珪素単結晶の製造方法。

【請求項3】 請求項1又は2において、上記成長結晶の端面の外周近傍においては、上記周壁から内方へ向けて上記エッチングガスを吐出することにより導入することを特徴とする炭化珪素単結晶の製造方法。

【請求項4】 請求項1～3のいずれか1項において、上記エッチングガスは、水素、ハロゲンガス、ハロゲン化水素のうち1種もしくは複数のガスを含有する混合ガスであることを特徴とする炭化珪素単結晶の製造方法。

【請求項5】 筒状の周壁を有する反応容器と、該反応容器内に配置された、炭化珪素単結晶よりなる基板結晶を支持する台座と、該台座に支持された上記基板結晶に向けて珪素を含有する珪素含有ガスと炭素を含有する炭素含有ガスを供給する反応ガス供給手段と、上記台座と上記周壁の間を通過して余剰の上記珪素含有ガス及び上記炭素含有ガスを排出する排気通路とを有する炭化珪素単結晶の製造装置において、上記基板結晶と上記台座との境界面の外周近傍に、炭化珪素をエッチングする効果のあるエッチングガスを導入する第1エッチングガス導入手段と、上記基板結晶上において成長した炭化珪素単結晶である成長結晶の端面の外周近傍に、炭化珪素をエッチングする効果のあるエッチングガスを導入する第2エッチングガス導入手段とを有することを特徴とする炭化珪素単結晶の製造装置。

【請求項6】 請求項5において、上記第1エッチングガス導入手段は、上記台座の外周面から外方に向かって開口する第1ガス噴出口を有することを特徴とする炭化珪素単結晶の製造装置。

【請求項7】 請求項5又は6において、上記第2エッチングガス導入手段は、上記反応容器の上記周壁の内周面から内方に向かって開口する第2ガス噴出口を有していることを特徴とする炭化珪素単結晶の製造装置。

【発明の詳細な説明】

【0001】

【技術分野】本発明は、CVD法（化学気相法）を利用して、高歩留まり、高効率で炭化珪素単結晶を成長させることができる炭化珪素単結晶の製造方法及び製造装置に関する。

【0002】

【従来技術】炭化珪素は、高耐圧、高電子移動度という特徴を有するため、パワーデバイス用半導体として期待されている。その基板となる炭化珪素単結晶を製造する方法としては、一般に、昇華法（改良レーリー法）と呼ばれる方法と、CVD法がある。上記改良レーリー法では、黒鉛製のつぼ内に炭化珪素原料を挿入すると共にこの原料部と対向するように種結晶（基板結晶）を黒鉛製のつぼの内壁に装着する。そして、原料部を2200～2400℃に加熱して昇華ガスを発生させ、原料部より数十～数百℃低温にした種結晶上において再結晶させることで炭化珪素単結晶を成長させる。

【0003】この改良レーリー法では、炭化珪素単結晶の成長に伴って炭化珪素原料が減少するため、成長させることができる量に限界がある。そして、たとえ、成長途中に原料を追加する手段をとったとしても、Si/Cが昇華する際にSi/C比が1を超える比で昇華するため、成長中に原料を追加するとつぼ内の昇華ガスの濃度や昇華速度が揺らぎ、結晶を連続的に高品質に成長させることの障害となってしまう。

【0004】一方、上記CVDによって炭化珪素をエピタキシャル成長させるものとしては、例えば特表平11-508531号公報に開示された方法がある。この方法は、円筒状の反応管（サセプタ）内に種結晶を配置し、SiやCを含有する原料ガスを供給して上記種結晶上において炭化珪素単結晶を成長させる方法である。この方法によれば、上記反応ガスの供給を連続的に行うことができるので、改良レーリー法に比べて、炭化珪素単結晶を長時間成長させ続けることができる。

【0005】

【解決しようとする課題】しかしながら、上記従来のCVD法では、結晶成長する箇所が、上記種結晶上に限らず、上記反応管の内周面上、あるいは上記原料ガスを排出する排出口近傍等の不要な箇所においても炭化珪素結晶が堆積し成長する。そのため、この不要な箇所での炭化珪素結晶の成長によって、原料ガスの供給が続けられず、種結晶上における炭化珪素単結晶の成長も途中で止めざるを得なくなってしまう。

【0006】この対策として、例えばWO 98/14644号公報に示されているように、反応容器の円周に沿って、反応には関与しないガス（He）を導入することによって排気口の目詰まりを防止する方法がある。しかしながら、この方法では、種結晶上で成長する炭化珪素単結晶（成長結晶）自身がエッチングされることが避

けられない。そのため、成長結晶の歩留まりが低下し、生産性が低下する。

【0007】本発明はかかる従来の問題点に鑑みてなされたもので、成長結晶の成長端における成長を確保した上で、成長結晶の成長端面以外の不要な箇所での炭化珪素単結晶の堆積を抑制することができ、成長結晶の長時間にわたる成長が可能な炭化珪素単結晶の製造方法及び製造装置を提供しようとするものである。

【0008】

【課題の解決手段】第1の発明は、筒状の周壁を有する反応容器内に、炭化珪素単結晶よりなる基板結晶を支持する台座を配置し、該台座に支持された上記基板結晶に向けて珪素を含有する珪素含有ガスと炭素を含有する炭素含有ガスを供給して上記基板結晶上において炭化珪素単結晶を成長させると共に、上記台座と上記周壁の間を通過して余剰の上記珪素含有ガス及び上記炭素含有ガスを排出する炭化珪素単結晶の製造方法において、上記基板結晶と上記台座との境界面の外周近傍と、上記基板結晶上において成長した炭化珪素単結晶である成長結晶の成長端面の外周近傍に、炭化珪素をエッチングする効果のあるエッチングガスを導入することを特徴とする炭化珪素単結晶の製造方法にある（請求項1）。

【0009】本発明においては、上記のごとく、上記基板結晶と上記台座との境界面の外周近傍と、上記基板結晶上において成長した炭化珪素単結晶である成長結晶の成長端面の外周近傍という少なくとも2箇所に、上記エッチングガスを導入する。これにより、上記成長結晶をほとんどエッチングすることなく、上記台座や周壁及び排気系統への炭化珪素単結晶の堆積を抑制することができる。

【0010】すなわち、上記成長結晶の成長端面に到達した上記珪素含有ガス及び炭素含有ガスは、基板結晶あるいはその上に既に形成された成長結晶の成長端面上において炭化珪素単結晶を成長させる。そして、この結晶の成長に寄与できなかった余剰のガスは、上記成長結晶と上記周壁との間を通過して排気されていく。このとき、本発明では、上記2箇所からのエッチングガスの導入により、上記余剰のガスの反応性を2段階で変化させることができる。

【0011】まず、上記基板結晶上において成長した炭化珪素単結晶である成長結晶の成長端面の外周近傍に、上記エッチングガスを導入する。これにより、未だ反応性を有する余剰の珪素含有ガス及び炭素含有ガスの混合ガスの状態が、珪素と炭素が過飽和で反応性の高い状態から、過飽和度がゼロの状態に近づくように、上記エッチングガスが作用する。これにより、上記余剰のガスとエッチングガスの混合ガスは、新たな結晶成長を抑制し、かつ、既に得られている成長結晶をエッチングしてしまうことを抑制する状態に変化する。

【0012】次に、上記混合ガスは、成長結晶の側面を

通過した後、上記基板結晶と上記台座との境界面の外周近傍において、さらに導入されたエッチングガスと混ざる。これにより、既に過飽和状態がゼロに近づいていた上記混合ガスは、さらに導入されたエッチングガスによって十分に未飽和の状態に変化し、また、上記エッチングガスによって炭化珪素結晶をエッチングする能力を有するものとなる。そのため、この段階における上記混合ガスは、新たな炭化珪素結晶の成長を確実に抑制し、かつ、新たに炭化珪素結晶が成長堆積しようとしても、上記エッチングガスのエッチング力によってその堆積を妨げる性質を有するものとなる。

【0013】このように、本発明においては、上記2段階のエッチングガスの導入を行うことによって、珪素含有ガスと炭素含有ガスの上記過飽和度の解消と、未飽和化とを段階的に行うことができる。これにより、上記成長結晶のエッチングを防止しつつ、排気系統への炭化珪素単結晶の堆積を防止することができる。それ故、成長結晶の長時間にわたる成長を可能にすることができる。また、必要に応じて、多段階のエッチングガス導入を行うことによって、さらに制御性の優れた結晶成長が可能となる。

【0014】第2の発明は、筒状の周壁を有する反応容器と、該反応容器内に配置された、炭化珪素単結晶よりなる基板結晶を支持する台座と、該台座に支持された上記基板結晶に向けて珪素を含有する珪素含有ガスと炭素を含有する炭素含有ガスを供給する反応ガス供給手段と、上記台座と上記周壁の間を通過して余剰の上記珪素含有ガス及び上記炭素含有ガスを排出する排気通路とを有する炭化珪素単結晶の製造装置において、上記基板結晶と上記台座との境界面の外周近傍に、炭化珪素をエッチングする効果のあるエッチングガスを導入する第1エッチングガス導入手段と、上記基板結晶上において成長した炭化珪素単結晶である成長結晶の端面の外周近傍に、炭化珪素をエッチングする効果のあるエッチングガスを導入する第2エッチングガス導入手段とを有することを特徴とする炭化珪素単結晶の製造装置にある（請求項5）。

【0015】本発明の製造装置においては、上記第1及び第2エッチングガス導入手段を有している。そのため、上記エッチングガスの導入を、2段階で行うことができる。それ故、上記のごとく優れた製造方法を実行することができ、炭化珪素単結晶の成長結晶の長時間にわたる成長を可能にすることができる。

【0016】

【発明の実施の形態】上記第1の発明（請求項1）において、上記基板結晶と上記台座との境界面の外周近傍においては、上記台座の内部から外部へ向けて上記エッチングガスを吐出することにより導入することが好ましい（請求項2）。この場合には、上記台座が移動する場合でも常にエッチングガスの導入位置を一定にすることが

でき、安定したエッチング効果が得られる。

【0017】また、上記成長結晶の端面の外周近傍においては、上記周壁から内方へ向けて上記エッチングガスを吐出することにより導入することが好ましい（請求項3）。この場合には、上記成長結晶の端面の外周へのエッチングガスの供給を成長端の位置に応じて容易に調整することができる。

【0018】また、上記エッチングガスは、水素、ハロゲンガス、ハロゲン化水素のうち1種もしくは複数のガスを含有する混合ガスであることが好ましい（請求項4）。これらのガスを用いれば、上記珪素含有ガス及び炭素含有ガスにおける珪素と炭素の過飽和度の調整を容易に行うことができると共に、炭化珪素単結晶のエッチング効果を容易に得ることができる。

【0019】上記第2の発明（請求項5）においては、上記第1エッチングガス導入手段は、上記台座の外周面から外方に向かって開口する第1ガス噴出口を有することが好ましい（請求項6）。これにより、エッチングガスの導入経路を上記台座の内部に設けることができ、エッチングガスの導入位置を一定にするための設備構造を簡単にすることができる。

【0020】また、上記第2エッチングガス導入手段は、上記反応容器の上記周壁の内周面から内方に向かって開口する第2ガス噴出口を有していることが好ましい（請求項7）。この場合には、エッチングガスの導入経路を上記周壁の外方に設けることができ、エッチングガスの導入位置を一定にするための設備構造を簡単にすることができる。

【0021】

【実施例】（実施例1）本発明の炭化珪素単結晶の製造方法及び製造装置に係る実施例につき、図1を用いて説明する。本例の炭化珪素単結晶の製造装置1は、図1に示すごとく、円筒状の周壁20を有する反応容器2と、該反応容器2内に配置された、炭化珪素単結晶よりなる基板結晶7を支持する台座3と、該台座3に支持された上記基板結晶7に向けて珪素を含有する珪素含有ガス81と炭素を含有する炭素含有ガス82を供給する反応ガス供給手段と、上記台座3と上記周壁20の間を通過して余剰の上記珪素含有ガス及び上記炭素含有ガスを排出する排気通路23とを有する。

【0022】上記基板結晶7と上記台座3との境界面37の外周近傍に、炭化珪素をエッチングする効果のあるエッチングガス9を導入する第1エッチングガス導入手段41と、基板結晶7上において成長した炭化珪素単結晶である成長結晶75の端面750の外周近傍に、炭化珪素をエッチングする効果のあるエッチングガス9を導入する第2エッチングガス導入手段42とを有する。

【0023】本例の第1エッチングガス導入手段41は、台座3の外周面30から外方に向かって開口する第1ガス噴出口410を有する。そして、この第1ガス噴

出口410に通じるように、台座3の内部にはエッチングガス導入管415を設け、これに図示しないエッチングガス供給源を接続してある。また、上記台座3は、成長結晶75の成長に応じて上昇するように構成されている。そして上記第1ガス噴出口410は、もちろん、台座3の上昇と共に上昇する。

【0024】また、本例の第2エッチングガス導入手段42は、反応容器2の上記周壁20の内周面から内方に向かって開口する第2ガス噴出口420を有している。この第2ガス噴出口420は、成長結晶75の成長端面750の外周に向いて開口している。上記第2ガス噴出口420に通じるエッチングガス導入管425は、図示しないエッチングガス供給源を接続されている。

【0025】また、本例の製造装置1は、上記反応容器2の周囲を石英管15により覆い、さらにその外方における上記反応容器2に対応する部分には、加熱用の高周波コイル18を設置してある。そして、上記石英管15の下端には上記珪素含有ガス81及び炭素含有ガス82を供給する供給管を備えた下蓋（図示略）が設けられ、石英管15の上端には、各種ガスを排気するための排気口を備えた上蓋（図示略）が設けられている。

【0026】また、本例では、上記基板結晶7として、4H-SiC単結晶基板を用い、上記台座3の下端に配設した。また、上記珪素含有ガス81としてはSiH₄ガスを、上記炭素含有ガス82としてはC₃H₈を用いた。そしてこれらのガス81、82を図1に示すごとくは反応容器2下方から導入し、基板結晶7上でSiCを成長させる。

【0027】成長に使われなかったガス及び原料ガスが分解して生成したH₂ガスは、成長結晶75の側面を通過して排気口へと向かう。ここで、本例の製造装置1は、上記第1エッチングガス導入手段41及び第2エッチング手段42を有している。これらを利用して、基板結晶7と台座3との境界面37の外周近傍へは、上記第1エッチングガス導入手段41からエッチングガス9を導入する。また、基板結晶7上において成長した炭化珪素単結晶である成長結晶75の成長端面750の外周近傍には、上記第2エッチングガス導入手段42からエッチングガス9を導入する。

【0028】これにより、エッチングガス9は、成長端面レベルでは外周方向から、基板結晶と台座との界面レベルでは台座から導入されるので、結晶成長とともに台座3を移動させても相対的な導入位置が変化せず、定常的なエッチングガス導入が行える。なお、エッチングガスの導入方向は中心軸方向（径方向）でも、接線方向（スワール方向）でも良い。

【0029】本例の成長条件は、基板温度2000～2500℃、SiH₄流量＝50～800sccm、C₃H₈流量＝10～300sccm、SiH₄、C₃H₈のキャリアガスとしてのAr＝1～10SLM、圧力＝100

～400 Torrであり、成長速度は0.5～3 mm/hである。また、上記エッチングガス9としては、H₂を用い、上記第2エッチングガス導入手段42においては5～10 SLM、第1エッチングガス導入手段41においては10～30 SLM導入した。

【0030】本例では、30時間の成長を実施した結果、成長結晶75の側壁における大きなエッチングは見られず、また、排気口においても結晶の堆積は見られなかったため、長時間にわたる結晶成長が可能であることが確認できた。この結果から、上記2つのエッチングガス導入手段41、42を用いて2段階のエッチングガス導入を行うことが、非常に有効であることがわかる。そして、この2段階のエッチングガス導入による作用効果は次のように考えられる。

【0031】すなわち、基板結晶7上において成長した炭化珪素単結晶である成長結晶75の成長端面750の外周近傍に、エッチングガス9を導入することにより、未だ反応性を有する余剰の珪素含有ガス81及び炭素含有ガス82の混合ガスの状態が、珪素と炭素の過飽和度が高く、結晶成長の進む状態から、過飽和度がゼロの状態に近づく。これにより、上記余剰のガスとエッチングガスの混合ガスは、新たな結晶成長を抑制し、かつ、既に得られている成長結晶をエッチングしてしまうことをも抑制する状態に変化する。

【0032】次に、上記混合ガスは、成長結晶75の側面を通過した後、上記基板結晶7と台座3との境界面37の外周近傍において、さらに導入されたエッチングガス9と混ざる。これにより、既に過飽和状態がゼロに近づいていた上記混合ガスは、さらに導入されたエッチングガス9によって十分に未飽和の状態に変化し、また、上記エッチングガス9によって炭化珪素結晶をエッチングする能力をも有するものとなる。そのため、この段階における上記混合ガスは、新たな炭化珪素結晶の成長を確実に抑制し、かつ、新たに炭化珪素結晶が堆積しようとしても、上記エッチングガスのエッチング力によってその堆積を確実に妨げる。

【0033】このように、本例においては、上記2段階

のエッチングガス9の導入を行うことによって、珪素含有ガス81と炭素含有ガス82の上記過飽和の解消と、未飽和化とを段階的に行うことができる。これにより、上記成長結晶75のエッチングを防止しつつ、排気系統への炭化珪素結晶の堆積を防止することができたのだと考えられる。

【0034】(実施例2) 本例では、実施例1と同様な構成の製造装置を用い、エッチングガス9としてHClを用いた点だけを変更した。この場合もエッチングガスとしてH₂を用いた場合と同様に成長結晶75の側壁における大きなエッチングは見られず、また、排気口においても結晶の堆積は見られなかった。そのため、多段にエッチングガス9を導入することにより、長時間の成長が可能であることが確認できた。

【0035】(比較例) 本比較例では、実施例1における、第1エッチングガス導入手段41によるエッチングガス9の導入を中止し、第2エッチングガス導入手段42からのみエッチングガス9を導入し、その他は実施例1と同様にして成長実験を行った。その結果、本比較例においては、成長時間の進行とともに台座側壁部にSiCが堆積し、排気口が閉塞を始めた。

【図面の簡単な説明】

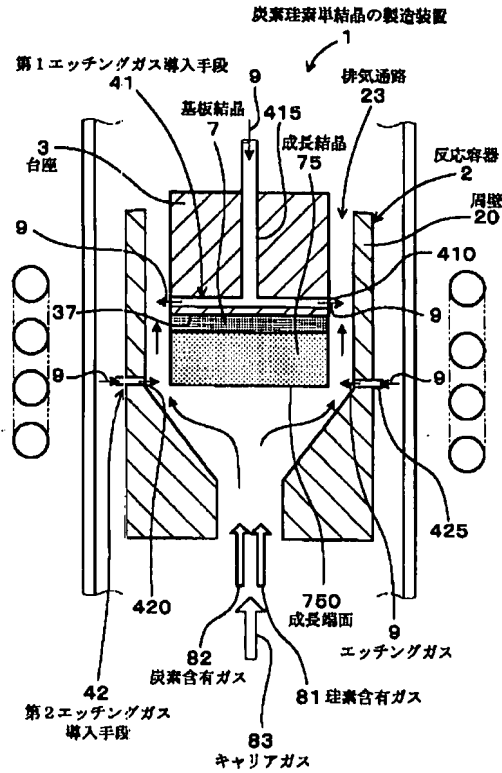
【図1】実施例1における、炭化珪素単結晶の製造装置の構成を示す説明図。

【符号の説明】

- 1... 炭化珪素単結晶の製造装置、
- 15... 石英管、
- 2... 反応容器、
- 20... 周壁、
- 3... 台座、
- 41... 第1エッチングガス導入手段、
- 42... 第2エッチングガス導入手段、
- 7... 基板結晶(種結晶)、
- 81... 珪素含有ガス、
- 82... 炭素含有ガス、
- 83... キャリアガス、
- 9... エッチングガス、

【図1】

(図1)



フロントページの続き

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1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. **** shows the word which can not be translated.
3. In the drawings, any words are not translated.

TECHNICAL FIELD

[Field of the Invention] This invention relates to the manufacture approach of the high yield and the silicon carbide monocrystal into which it can be efficient and silicon carbide monocrystal can be grown up, and a manufacturing installation using a CVD method (chemistry gaseous-phase method).

PRIOR ART

[Description of the Prior Art] Since it has the description of high pressure-proofing and high electron mobility, silicon carbide is expected as a semiconductor for power devices. Generally as an approach of manufacturing silicon carbide single **** used as the substrate, there are an approach called the sublimating method (amelioration Rayleigh law) and a CVD method. By the describing [above] amelioration Rayleigh method, while inserting a silicon carbide raw material into the crucible made from a graphite, the wall of the crucible made from a graphite is equipped with seed crystal (substrate crystal) so that it may counter with this raw material section. And the raw material section is heated at 2200-2400 degrees C, sublimation gas is generated, and silicon carbide monocrystal is grown up by making it recrystallize on the seed crystal made into a ten number - hundreds of degrees-C low temperature from the raw material section.

[0003] By this amelioration Rayleigh method, in order that silicon carbide raw materials may decrease in number with growth of silicon carbide monocrystal, a limitation is in the amount which can be grown up. And since a Si/C ratio will sublimate by the ratio exceeding 1 in case SiC sublimates even if it takes means to add a raw material in the middle of growth, the concentration and the sublimation rate of sublimation gas in the end crater which adds a raw material during growth will become the failure of growing up fluctuation and a crystal into high quality continuously.

[0004] On the other hand, the approach indicated by the Patent Publication

Heisei No. 508531 [11 to] official report, for example is one of those to which epitaxial growth of the silicon carbide is carried out by Above CVD. This approach is an approach of arranging seed crystal, supplying the material gas containing Si or C in a cylinder-like coil (susceptor), and growing up silicon carbide monocrystal on the above-mentioned seed crystal. since the above-mentioned reactant gas can be supplied continuously according to this approach -- amelioration Rayleigh -- growing up silicon carbide monocrystal can be continued for a long time compared with law

10 TECHNICAL PROBLEM

[Problem(s) to be Solved] However, in the above-mentioned conventional CVD method, also in a part unnecessary [near / where the part which carries out crystal growth discharges the above-mentioned material gas on the inner skin of not only the above-mentioned seed crystal top but the above-mentioned coil / the exhaust port], a silicon carbide crystal accumulates and it grows up. Therefore, by the silicon carbide crystal growth in this unnecessary part, supply of material gas is not continued, but growth of the silicon carbide monocrystal on seed crystal will also obtain a stop colander on the way, and will be lost.

[0006] As this cure, it is WO. There is a method of preventing the blinding of an exhaust port by introducing into a reaction the gas (helium) which does not involve in accordance with the periphery of a reaction container as shown in the No. 98/14644 official report. However, by this approach, it is not avoided that the silicon carbide monocrystal (growth crystal) itself which grows on seed crystal is etched. Therefore, the yield of a growth crystal falls and productivity falls.

[0007] After being made in view of this conventional trouble and securing the growth in a growth crystal growth edge, this invention can control deposition of the silicon carbide monocrystal in unnecessary parts other than a growth crystal growth end face, and tends to offer the manufacture approach of the silicon carbide monocrystal in which the growth covering the long time of a growth crystal is possible, and a manufacturing installation.

35 MEANS

[Means for Solving the Problem] In the reaction container which has a tubed peripheral wall, the 1st invention arranges the plinth which supports the substrate crystal which consists of silicon carbide monocrystal, supplies the silicon content gas which contains silicon towards the above-mentioned substrate crystal supported by this plinth, and the carbon content gas containing carbon, and sets them on the above-mentioned substrate crystal. In the manufacture approach of the silicon carbide monocrystal which passes through between the above-mentioned plinth and the above-mentioned peripheral walls, and discharges the excessive above-mentioned silicon content gas and the above-mentioned carbon content gas while growing up silicon carbide monocrystal In the above-mentioned substrate crystal top near the periphery of the interface of the above-mentioned substrate crystal and the above-mentioned plinth It is in the manufacture approach of the silicon carbide monocrystal characterized by introducing etching gas with the effectiveness which etches silicon carbide near the periphery of the grown-up growth crystal growth end face which is silicon carbide monocrystal (claim 1).

[0009] In this invention, the above-mentioned etching gas is introduced into at least two saying near the periphery of the growth crystal growth end face which grew on the above-mentioned substrate crystal and which is silicon carbide monocrystal near the periphery of the interface of the above-mentioned substrate crystal and the above-mentioned plinth places like the above. Thereby, deposition of the silicon carbide monocrystal to the above-mentioned plinth, a peripheral wall, and an exhaust system can be controlled, without etching most above-mentioned growth crystals.

[0010] That is, the above-mentioned silicon content gas and carbon content gas which reached the above-mentioned growth crystal growth end face grow up silicon carbide monocrystal on the growth crystal growth end face already formed a substrate crystal or on it. And the gas of the surplus which was not able to contribute to this crystal growth passes through between the above-mentioned growth crystal and the above-mentioned peripheral walls, and is exhausted. At this time, the reactivity of the gas of the above-mentioned surplus can be changed by installation of the etching gas from [above-mentioned] two places by this invention in two steps.

[0011] First, the above-mentioned etching gas is introduced near the periphery of the growth crystal growth end face which grew on the above-mentioned substrate crystal and which is silicon carbide monocrystal. The above-mentioned etching gas acts so that a degree of supersaturation may

approach [the condition of the mixed gas of the silicon content gas of the surplus which still has reactivity, and carbon content gas / silicon and carbon] a zero state from a reactant high condition by supersaturation by this. This changes to the condition of also controlling the gas of the above-mentioned surplus and the mixed gas of etching gas etching the growth crystal which controls new crystal growth and has already been obtained.

[0012] Next, after the above-mentioned mixed gas passes through the side face of a growth crystal, it is mixed with the etching gas introduced further [near the periphery of the interface of the above-mentioned substrate crystal and the above-mentioned plinth]. Thereby, the above-mentioned mixed gas by which the supersaturation condition had already approached zero also has the capacity which fully changes with the etching gas introduced further to the condition of unsaturation, and etches a silicon carbide crystal with the above-mentioned etching gas. Therefore, the above-mentioned mixed gas in this phase controls new silicon carbide crystal growth certainly, and even if a silicon carbide crystal newly tends to carry out growth deposition, it has the property which bars that deposition according to the etching force of the above-mentioned etching gas.

[0013] Thus, in this invention, dissolution of the above-mentioned degree of supersaturation of silicon content gas and carbon content gas and unsaturation-ization can be gradually performed by introducing the two steps of above-mentioned etching gas. Thereby, deposition of the silicon carbide monocrystal to an exhaust system can be prevented, preventing etching of the above-mentioned growth crystal. So, growth covering the long time of a growth crystal can be enabled. Moreover, the crystal growth which was further excellent in the controllability becomes possible by performing etching gas installation of a multistage story if needed.

[0014] The plinth which supports the substrate crystal with which the 2nd invention has been arranged in the reaction container which has a tubed peripheral wall, and this reaction container, and which consists of silicon carbide monocrystal, A reactant gas supply means to supply the silicon content gas which contains silicon towards the above-mentioned substrate crystal supported by this plinth, and the carbon content gas containing carbon, In the manufacturing installation of silicon carbide monocrystal which has the flueway which passes through between the above-mentioned plinth and the above-mentioned peripheral walls, and discharges the excessive above-mentioned silicon content gas and the above-mentioned carbon

content gas A 1st etching gas installation means to introduce the etching gas which has the effectiveness which etches silicon carbide near the periphery of the interface of the above-mentioned substrate crystal and the above-mentioned plinth, It is in the manufacturing installation of the silicon carbide
 5 monocrystal characterized by having a 2nd etching gas installation means to introduce etching gas with the effectiveness which etches silicon carbide, near the periphery of the end face of the growth crystal which is silicon carbide monocrystal which grew on the above-mentioned substrate crystal (claim 5).

10 [0015] In the manufacturing installation of this invention, it has the above-mentioned 1st and 2nd etching gas installation means. Therefore, the above-mentioned etching gas can be introduced in two steps. So, like the above, the outstanding manufacture approach can be performed and growth covering the long time of the growth crystal of silicon carbide monocrystal can be
 15 enabled.

[0016]

[Embodiment of the Invention] In the 1st above-mentioned invention (claim 1), it is [/ near the periphery of the interface of the above-mentioned substrate crystal and the above-mentioned plinth] desirable to introduce by carrying out
 20 the regurgitation of the above-mentioned etching gas towards the exterior from the interior of the above-mentioned plinth (claim 2). In this case, even when the above-mentioned plinth moves, the introductory location of etching gas can always be made regularity, and the stable etching effectiveness is acquired.

25 [0017] Moreover, it is [/ near the periphery of the end face of the above-mentioned growth crystal] desirable to introduce by carrying out the regurgitation of the above-mentioned etching gas towards the method of inside from the above-mentioned peripheral wall (claim 3). In this case, supply of the etching gas to the periphery of the end face of the above-mentioned
 30 growth crystal can be easily adjusted according to the location of a growth edge.

[0018] Moreover, it is desirable that the above-mentioned etching gas is hydrogen, halogen gas, and mixed gas that contains one sort or two or more gas among hydrogen halides (claim 4). If these gas is used, while being able
 35 to adjust the degree of supersaturation of the silicon in the above-mentioned silicon content gas and carbon content gas, and carbon easily, the etching effectiveness of silicon carbide monocrystal can be acquired easily.

[0019] In the 2nd above-mentioned invention (claim 5), it is desirable that the above-mentioned 1st etching gas installation means has the 1st gas port which carries out opening toward the method of outside from the peripheral face of the above-mentioned plinth (claim 6). Thereby, the introductory path of etching gas can be prepared in the interior of the above-mentioned plinth, and facility structure for making the introductory location of etching gas regularity can be simplified.

[0020] Moreover, it is desirable that the above-mentioned 2nd etching gas installation means has the 2nd gas port which carries out opening toward the method of inside from the inner skin of the above-mentioned peripheral wall of the above-mentioned reaction container (claim 7). In this case, the introductory path of etching gas can be prepared in a way outside the above-mentioned peripheral wall, and facility structure for making the introductory location of etching gas regularity can be simplified.

15

DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

20 [Field of the Invention] This invention relates to the manufacture approach of the high yield and the silicon carbide monocrystal into which it can be efficient and silicon carbide monocrystal can be grown up, and a manufacturing installation using a CVD method (chemistry gaseous-phase method).

[0002]

25 [Description of the Prior Art] Since it has the description of high pressure-proofing and high electron mobility, silicon carbide is expected as a semiconductor for power devices. Generally as an approach of manufacturing silicon carbide single **** used as the substrate, there are an approach called the sublimating method (amelioration Rayleigh law) and a CVD method. By
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35 generated, and silicon carbide monocrystal is grown up by making it recrystallize on the seed crystal made into a-ten number - hundreds of degrees-C low temperature from the raw material section.

[0003] By this amelioration Rayleigh method, in order that silicon carbide raw materials may decrease in number with growth of silicon carbide monocrystal, a limitation is in the amount which can be grown up. And since a Si/C ratio will sublime by the ratio exceeding 1 in case SiC sublimates even if it takes
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35 [0007] After being made in view of this conventional trouble and securing the growth in a growth crystal growth edge, this invention can control deposition of the silicon carbide monocrystal in unnecessary parts other than a growth

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[0008]

- 5 [Means for Solving the Problem] In the reaction container which has a tubed peripheral wall, the 1st invention arranges the plinth which supports the substrate crystal which consists of silicon carbide monocrystal, supplies the silicon content gas which contains silicon towards the above-mentioned substrate crystal supported by this plinth, and the carbon content gas
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- [0012] Next, after the above-mentioned mixed gas passes through the side face of a growth crystal, it is mixed with the etching gas introduced further [near the periphery of the interface of the above-mentioned substrate crystal and the above-mentioned plinth]. Thereby, the above-mentioned mixed gas by which the supersaturation condition had already approached zero also has the capacity which fully changes with the etching gas introduced further to the condition of unsaturation, and etches a silicon carbide crystal with the above-mentioned etching gas. Therefore, the above-mentioned mixed gas in this phase controls new silicon carbide crystal growth certainly, and even if a silicon carbide crystal newly tends to carry out growth deposition, it has the property which bars that deposition according to the etching force of the above-mentioned etching gas.
- [0013] Thus, in this invention, dissolution of the above-mentioned degree of supersaturation of silicon content gas and carbon content gas and unsaturation-ization can be gradually performed by introducing the two steps of above-mentioned etching gas. Thereby, deposition of the silicon carbide monocrystal to an exhaust system can be prevented, preventing etching of the above-mentioned growth crystal. So, growth covering the long time of a growth crystal can be enabled. Moreover, the crystal growth which was further excellent in the controllability becomes possible by performing etching gas installation of a multistage story if needed.
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15 [0015] In the manufacturing installation of this invention, it has the above-mentioned 1st and 2nd etching gas installation means. Therefore, the above-mentioned etching gas can be introduced in two steps. So, like the above, the outstanding manufacture approach can be performed and growth covering the long time of the growth crystal of silicon carbide monocrystal can be enabled.

20 [0016]
[Embodiment of the Invention] In the 1st above-mentioned invention (claim 1), it is [/ near the periphery of the interface of the above-mentioned substrate crystal and the above-mentioned plinth] desirable to introduce by carrying out the regurgitation of the above-mentioned etching gas towards the exterior
25 from the interior of the above-mentioned plinth (claim 2). In this case, even when the above-mentioned plinth moves, the introductory location of etching gas can always be made regularity, and the stable etching effectiveness is acquired.

[0017] Moreover, it is [/ near the periphery of the end face of the above-mentioned growth crystal] desirable to introduce by carrying out the regurgitation of the above-mentioned etching gas towards the method of inside from the above-mentioned peripheral wall (claim 3). In this case, supply of the etching gas to the periphery of the end face of the above-mentioned growth crystal can be easily adjusted according to the location of a growth edge.

[0018] Moreover, it is desirable that the above-mentioned etching gas is hydrogen, halogen gas, and mixed gas that contains one sort or two or more

gas among hydrogen halides (claim 4). If these gas is used, while being able to adjust the degree of supersaturation of the silicon in the above-mentioned silicon content gas and carbon content gas, and carbon easily, the etching effectiveness of silicon carbide monocrystal can be acquired easily.

- 5 [0019] In the 2nd above-mentioned invention (claim 5), it is desirable that the above-mentioned 1st etching gas installation means has the 1st gas port which carries out opening toward the method of outside from the peripheral face of the above-mentioned plinth (claim 6). Thereby, the introductory path of etching gas can be prepared in the interior of the above-mentioned plinth, and
10 facility structure for making the introductory location of etching gas regularity can be simplified.

- [0020] Moreover, it is desirable that the above-mentioned 2nd etching gas installation means has the 2nd gas port which carries out opening toward the method of inside from the inner skin of the above-mentioned peripheral wall of
15 the above-mentioned reaction container (claim 7). In this case, the introductory path of etching gas can be prepared in a way outside the above-mentioned peripheral wall, and facility structure for making the introductory location of etching gas regularity can be simplified.

[0021]

- 20 [Example] (Example 1) It explains using drawing 1 about the example concerning the manufacture approach of the silicon carbide monocrystal of this invention, and a manufacturing installation. The reaction container 2 which has the cylinder-like peripheral wall 20 as the manufacturing installation 1 of the silicon carbide monocrystal of this example is shown in drawing 1 ,
25 The plinth 3 which supports the substrate crystal 7 which has been arranged in this reaction container 2, and which consists of silicon carbide monocrystal, A reactant gas supply means to supply the silicon content gas 81 which contains silicon towards the above-mentioned substrate crystal 7 supported by this plinth 3, and the carbon content gas 82 containing carbon, It has the
30 flueway 23 which passes through between the above-mentioned plinth 3 and the above-mentioned peripheral walls 20, and discharges the excessive above-mentioned silicon content gas and the above-mentioned carbon content gas.

- [0022] Near the periphery of the interface 37 of the above-mentioned
35 substrate crystal 7 and the above-mentioned plinth 3, it has a 2nd etching gas installation means 42 introduce etching gas 9 with the effectiveness which etches silicon carbide, near the periphery of the end face 750 of the growth

crystal 75 which are a 1st etching gas installation means 41 to introduce etching gas 9 with the effectiveness which etches silicon carbide, and silicon carbide monocrystal which grew on the substrate crystal 7.

[0023] The 1st etching gas installation means 41 of this example has the 1st gas port 410 which carries out opening toward the method of outside from the peripheral face 30 of a plinth 3. And the etching gas installation tubing 415 is formed in the interior of a plinth 3, and the etching gas source of supply which is not illustrated to this is connected so that it may lead to this 1st gas port 410. Moreover, the above-mentioned plinth 3 is constituted so that it may go up according to growth of the growth crystal 75. And, of course, the 1st gas port 410 of the above goes up with a rise of a plinth 3.

[0024] Moreover, the 2nd etching gas installation means 42 of this example has the 2nd gas port 420 which carries out opening toward the method of inside from the inner skin of the above-mentioned peripheral wall 20 of the reaction container 2. Opening of this 2nd gas port 420 is carried out toward the periphery of the growth end face 750 of the growth crystal 75. The etching gas source of supply which does not illustrate the etching gas installation tubing 425 which leads to the 2nd gas port 420 of the above is connected.

[0025] Moreover, the manufacturing installation 1 of this example covers the perimeter of the above-mentioned reaction container 2 with a quartz tube 15, and has installed the high frequency coil 18 for heating in the part corresponding to the above-mentioned reaction container 2 in the method of the outside further. And the lower lid (illustration abbreviation) equipped with the supply pipe which supplies the above-mentioned silicon content gas 81 and carbon content gas 82 is formed in the lower limit of the above-mentioned quartz tube 15, and the top cover (illustration abbreviation) equipped with the exhaust port for exhausting various gas is prepared in the upper limit of a quartz tube 15.

[0026] Moreover, it arranged in the lower limit of the above-mentioned plinth 3 in this example, using 4 H-SiC single crystal substrate as the above-mentioned substrate crystal 7. Moreover, SiH₄ gas was used as the above-mentioned silicon content gas 81, and C₃H₈ were used as the above-mentioned carbon content gas 82. And profit which shows these gas 81 and 82 to drawing 1 is introduced from reaction container 2 lower part, and grows up SiC on the substrate crystal 7.

[0027] H₂ gas which the gas and material gas which were not used for growth decomposed and generated passes through the side face of the growth

crystal 75, and goes to an exhaust port. Here, the manufacturing installation 1 of this example has the above-mentioned 1st etching gas installation means 41 and the 2nd etching means 42. Near the periphery of the interface 37 of the substrate crystal 7 and a plinth 3, etching gas 9 is introduced from the

5 above-mentioned 1st etching gas installation means 41 using these.

Moreover, near the periphery of the growth end face 750 of the growth crystal 75 which is silicon carbide monocrystal which grew on the substrate crystal 7, etching gas 9 is introduced from the above-mentioned 2nd etching gas installation means 42.

10 [0028] Thereby, on growth end-face level, since etching gas 9 is introduced from a plinth on the interface level of a substrate crystal and a plinth from a periphery, even if it moves a plinth 3 with crystal growth, a relative introductory location does not change, but it can perform steady etching gas installation from it. In addition, the direction of a medial axis (the direction of a

15 path) or a tangential direction (the direction of a swirl) is sufficient as the introductory direction of etching gas.

[0029] the growth conditions of this example -- the substrate temperature of 2000-2500 degrees C, and SiH₄ flow rate = -- it is 50 - 800sccm, C₃H₈ flow-rate = 10 - 300sccm, SiH₄, Ar=1 - 10SLM as carrier gas of C₃H₈, and

20 pressure = 100 - 400Torr, and a growth rate is 0.5 - 3 mm/h. Moreover, in the above-mentioned 2nd etching gas installation means 42, 10-30SLM installation was carried out in 5-10SLM and the 1st etching gas installation means 41, using H₂ as the above-mentioned etching gas 9.

[0030] In this example, since big etching in the side attachment wall of the

25 growth crystal 75 was not seen and deposition of a crystal was not seen in the exhaust port as a result of carrying out growth of 30 hours, it has checked that the crystal growth covering long duration was possible. This result shows that it is very effective to perform two steps of etching gas installation using the two above-mentioned etching gas installation means 41 and 42. And the

30 operation effectiveness by two steps of this etching gas installation is considered as follows.

[0031] That is, a degree of supersaturation approaches a zero state by introducing etching gas 9 from the condition that the degree of supersaturation of silicon and carbon has the high condition of the mixed gas

35 of the silicon content gas 81 of the surplus which still has reactivity, and carbon content gas 82, and crystal growth progresses, near the periphery of the growth end face 750 of the growth crystal 75 which is silicon carbide

monocrystal which grew on the substrate crystal 7. This changes to the condition of also controlling the gas of the above-mentioned surplus and the mixed gas of etching gas etching the growth crystal which controls new crystal growth and has already been obtained.

5 [0032] Next, after the above-mentioned mixed gas passes through the side face of the growth crystal 75, it is mixed with the etching gas 9 introduced further [near the periphery of the interface 37 of the above-mentioned substrate crystal 7 and a plinth 3]. Thereby, the above-mentioned mixed gas by which the supersaturation condition had already approached zero also has
10 the capacity which fully changes with the etching gas 9 introduced further to the condition of unsaturation, and etches a silicon carbide crystal with the above-mentioned etching gas 9. Therefore, the above-mentioned mixed gas in this phase controls new silicon carbide crystal growth certainly, and even if a silicon carbide crystal newly tends to accumulate, it bars that deposition
15 certainly according to the etching force of the above-mentioned etching gas.

[0033] Thus, in this example, dissolution of the above-mentioned supersaturation of silicon content gas 81 and carbon content gas 82 and unsaturation-ization can be gradually performed by introducing the two steps of above-mentioned etching gas 9. Thereby, it is thought that deposition of
20 the silicon carbide crystal to an exhaust system was able to be prevented, preventing etching of the above-mentioned growth crystal 75.

[0034] (Example 2) In this example, only the point using HCl as etching gas 9 was changed using the manufacturing installation of the same configuration as an example 1. Big etching in the side attachment wall of the growth crystal
25 75 was not seen like the case where H₂ is used as etching gas also in this case, and deposition of a crystal was not seen in the exhaust port. Therefore, it has checked that growth of long duration was possible by introducing etching gas 9 into multistage.

[0035] (Example of a comparison) In this example of a comparison, the
30 installation of the etching gas 9 by the 1st etching gas installation means 41 in an example 1 was stopped, etching gas 9 was introduced only from the 2nd etching gas installation means 42, and others conducted the growth experiment like the example 1. Consequently, in this example of a comparison, SiC accumulated on the plinth side-attachment-wall section with
35 advance of growth time amount, and the exhaust port began lock out.

CLAIMS

[Claim(s)]

- 5 [Claim 1] The silicon content gas which contains silicon towards the above-mentioned substrate crystal which has arranged the plinth which supports the substrate crystal which consists of silicon carbide monocrystal, and was supported by this plinth in the reaction container which has a tubed peripheral wall, and the carbon content gas containing carbon are supplied. In the
- 10 manufacture approach of the silicon carbide monocrystal which passes through between the above-mentioned plinth and the above-mentioned peripheral walls, and discharges the excessive above-mentioned silicon content gas and the above-mentioned carbon content gas while growing up silicon carbide monocrystal on the above-mentioned substrate crystal The
- 15 manufacture approach of the silicon carbide monocrystal characterized by introducing etching gas with the effectiveness which etches silicon carbide near the periphery of the growth crystal growth end face which grew on the above-mentioned substrate crystal, and which is silicon carbide monocrystal near the periphery of the interface of the above-mentioned substrate crystal
- 20 and the above-mentioned plinth.
- [Claim 2] The manufacture approach of the silicon carbide monocrystal characterized by introducing in claim 1 [near the periphery of the interface of the above-mentioned substrate crystal and the above-mentioned plinth] by carrying out the regurgitation of the above-mentioned etching gas towards the
- 25 exterior from the interior of the above-mentioned plinth.
- [Claim 3] The manufacture approach of the silicon carbide monocrystal characterized by introducing [near the periphery of the end face of the above-mentioned growth crystal] in claim 1 or 2 by carrying out the regurgitation of the above-mentioned etching gas towards the method of inside from the
- 30 above-mentioned peripheral wall.
- [Claim 4] It is the manufacture approach of the silicon carbide monocrystal characterized by being the mixed gas in which the above-mentioned etching gas contains one sort or two or more gas among hydrogen, halogen gas, and hydrogen halide in any 1 term of claims 1-3.
- 35 [Claim 5] The reaction container which has a tubed peripheral wall, and the plinth which supports the substrate crystal which has been arranged in this reaction container, and which consists of silicon carbide monocrystal, A

reactant gas supply means to supply the silicon content gas which contains silicon towards the above-mentioned substrate crystal supported by this plinth, and the carbon content gas containing carbon, In the manufacturing installation of silicon carbide monocrystal which has the flueway which passes

5 through between the above-mentioned plinth and the above-mentioned peripheral walls, and discharges the excessive above-mentioned silicon content gas and the above-mentioned carbon content gas A 1st etching gas installation means to introduce the etching gas which has the effectiveness which etches silicon carbide near the periphery of the interface of the above-mentioned substrate crystal and the above-mentioned plinth, The
10 manufacturing installation of the silicon carbide monocrystal characterized by having a 2nd etching gas installation means to introduce etching gas with the effectiveness which etches silicon carbide, near the periphery of the end face of the growth crystal which is silicon carbide monocrystal which grew on the
15 above-mentioned substrate crystal.

[Claim 6] It is the manufacturing installation of the silicon carbide monocrystal characterized by having the 1st gas port which carries out opening of the above-mentioned 1st etching gas installation means toward the method of outside in claim 5 from the peripheral face of the above-mentioned plinth.

20 [Claim 7] It is the manufacturing installation of the silicon carbide monocrystal characterized by having the 2nd gas port which carries out opening of the above-mentioned 2nd etching gas installation means toward the method of inside in claim 5 or 6 from the inner skin of the above-mentioned peripheral wall of the above-mentioned reaction container.

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DRAWINGS

[Drawing 1]

(図1)

